

FORM PTO-1390 (Modified)  
(REV 11-2000)

U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

ATTORNEY'S DOCKET NUMBER

**TRANSMITTAL LETTER TO THE UNITED STATES  
DESIGNATED/ELECTED OFFICE (DO/EO/US)  
CONCERNING A FILING UNDER 35 U.S.C. 371**

**216527US0PCT**

U.S. APPLICATION NO. (IF KNOWN, SEE 37 CFR

**10/019427**

INTERNATIONAL APPLICATION NO.  
**PCT/JP00/04359**

INTERNATIONAL FILING DATE  
**30 JUNE 2000**

PRIORITY DATE CLAIMED  
**30 JUNE 1999**

TITLE OF INVENTION

**WATER-IN-OIL TYPE EMULSIFIED FAT AND/OR OIL COMPOSITION**

APPLICANT(S) FOR DO/EO/US

**Kenji MASUI, et al.**


Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This is an express request to begin national examination procedures (35 U.S.C. 371(f)). The submission must include items (5), (6), (9) and (24) indicated below.
4. ☒ The US has been elected by the expiration of 19 months from the priority date (Article 31).
5. ☒ A copy of the International Application as filed (35 U.S.C. 371 (c) (2))
  - a. ☐ is attached hereto (required only if not communicated by the International Bureau).
  - b. ☒ has been communicated by the International Bureau.
  - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☐ An English language translation of the International Application as filed (35 U.S.C. 371(c)(2)).
  - a. ☐ is attached hereto.
  - b. ☐ has been previously submitted under 35 U.S.C. 154(d)(4).
7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371 (c)(3))
  - a. ☐ are attached hereto (required only if not communicated by the International Bureau).
  - b. ☐ have been communicated by the International Bureau.
  - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
  - d. ☒ have not been made and will not be made.
8. ☐ An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☒ An oath or declaration of the inventor(s) (35 U.S.C. 371 (c)(4)).
10. ☐ An English language translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371 (c)(5)).
11. ☒ A copy of the International Preliminary Examination Report (PCT/IPEA/409).
12. ☒ A copy of the International Search Report (PCT/ISA/210).

**Items 13 to 20 below concern document(s) or information included:**

13. ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
14. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
15. ☒ A **FIRST** preliminary amendment.
16. ☐ A **SECOND** or **SUBSEQUENT** preliminary amendment.
17. ☐ A substitute specification.
18. ☐ A change of power of attorney and/or address letter.
19. ☐ A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 U.S.C. 1.821 - 1.825.
20. ☐ A second copy of the published international application under 35 U.S.C. 154(d)(4).
21. ☐ A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4).
22. ☐ Certificate of Mailing by Express Mail
23. ☒ Other items or information:

**Request for Consideration of Documents in International Search Report  
Notice of Priority / PCT/IB/304 / PCT/IB/308**

U.S. APPLICATION NO. (IF KNOWN, SEE 37 CFR 10/019427)		INTERNATIONAL APPLICATION NO. PCT/JP00/04359		ATTORNEY'S DOCKET NUMBER 216527US0PCT	
24. The following fees are submitted: <b>BASIC NATIONAL FEE ( 37 CFR 1.492 (a) (1) - (5)) :</b> <input type="checkbox"/> Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO ..... \$1040.00 <input checked="" type="checkbox"/> International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO ..... \$890.00 <input type="checkbox"/> International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO ..... \$740.00 <input type="checkbox"/> International preliminary examination fee (37 CFR 1.482) paid to USPTO but all claims did not satisfy provisions of PCT Article 33(1)-(4) ..... \$710.00 <input type="checkbox"/> International preliminary examination fee (37 CFR 1.482) paid to USPTO and all claims satisfied provisions of PCT Article 33(1)-(4) ..... \$100.00 <b>ENTER APPROPRIATE BASIC FEE AMOUNT =</b> \$890.00				<b>CALCULATIONS PTO USE ONLY</b>	
Surcharge of \$130.00 for furnishing the oath or declaration later than months from the earliest claimed priority date (37 CFR 1.492 (e)). <input type="checkbox"/> 20 <input type="checkbox"/> 30				\$0.00	
CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE		
Total claims	7 - 20 =	0	x \$18.00	\$0.00	
Independent claims	3 - 3 =	0	x \$84.00	\$0.00	
Multiple Dependent Claims (check if applicable). <input type="checkbox"/>				\$0.00	
<b>TOTAL OF ABOVE CALCULATIONS =</b>				\$890.00	
<input type="checkbox"/> Applicant claims small entity status. See 37 CFR 1.27). The fees indicated above are reduced by 1/2.				\$0.00	
<b>SUBTOTAL =</b>				\$890.00	
Processing fee of \$130.00 for furnishing the English translation later than months from the earliest claimed priority date (37 CFR 1.492 (f)). <input type="checkbox"/> 20 <input type="checkbox"/> 30 +				\$0.00	
<b>TOTAL NATIONAL FEE =</b>				\$890.00	
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31) (check if applicable). <input type="checkbox"/>				\$0.00	
<b>TOTAL FEES ENCLOSED =</b>				\$890.00	
				Amount to be: refunded	\$
				charged	\$
a. <input checked="" type="checkbox"/> A check in the amount of \$890.00 to cover the above fees is enclosed. b. <input type="checkbox"/> Please charge my Deposit Account No. _____ in the amount of _____ to cover the above fees. A duplicate copy of this sheet is enclosed. c. <input checked="" type="checkbox"/> The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 15-0030 A duplicate copy of this sheet is enclosed. d. <input type="checkbox"/> Fees are to be charged to a credit card. <b>WARNING:</b> Information on this form may become public. <b>Credit card information should not be included on this form.</b> Provide credit card information and authorization on PTO-2038.					
<b>NOTE:</b> Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.					
SEND ALL CORRESPONDENCE TO:					
 <b>22850</b>  Surinder Sachar Registration No. 34,423 (703) 413-3000			_____ SIGNATURE  Norman F. Oblon NAME  24,618 REGISTRATION NUMBER  Dec. 31 2001 DATE		

216527US-0PCT

IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF :  
KENJI MASUI ET AL : ATTN: APPLICATION DIVISION  
SERIAL NO: NEW U.S. PCT APPLN :  
(Based on PCT/JP00/04359)  
FILED: HEREWITH :  
FOR: WATER-IN-OIL TYPE :  
EMULSIFIED FAT AND/OR  
OIL COMPOSITION

PRELIMINARY AMENDMENT

ASSISTANT COMMISSIONER FOR PATENTS  
WASHINGTON, D.C. 20231

SIR:

Prior to examination on the merits, please amend the above-identified application as follows.

IN THE CLAIMS

Please cancel Claims 1-6.

Please add the following new claims:

7. (New) A method comprising adding a demulsifier to an emulsion composition, said demulsifier acting to reverse at least 30% of a water-in-oil phase of said emulsion composition within one minute after said emulsion composition has been introduced into water at 36°C,

wherein said composition comprises an aqueous phase based on water and an oil phase, said oil phase comprising at least one component selected from the group consisting of fats, oils, or mixtures thereof,

wherein said oil phase comprises at least one diglyceride, oil, fat or mixture thereof and at least one flavor component, and

said oil phase is at least 15% by weight based on a total weight of said composition.

8. (New) The method as claimed in Claim 7, wherein said oil phase comprises 15-90% by weight of at least one diglyceride and 85-10% by weight of at least one triglyceride, and

said composition comprises 0.05-20% by weight of the flavor component and from 0.01-5% by weight of the demulsifier.

9. (New) The method as claimed in Claim 7, wherein a weight ratio of the aqueous phase to the oil phase ranges between 80:20 and 15:85.

10. (New) The method as claimed in Claim 7, wherein the demulsifier is at least one member selected from the group consisting of a polyglycerol fatty acid ester having HLB of 7 or more, a water soluble decomposed protein, lysolecithin having an HLB of 8 or more, a sucrose fatty acid ester having an HLB of 5 or more, a monoglyceride organic ester having an HLB of 8 or more and a sorbitan fatty acid ester having an HLB of 8 or more.

11. (New) A water-in-oil emulsion composition comprising an aqueous phase based on water and an oil phase, said oil phase comprising at least one member selected from the group consisting of fats, oils or mixtures thereof and comprises 15-90% by weight of at least one diglyceride and 85-10% by weight of at least one triglyceride, and

said composition comprises 0.01-5% by weight of a demulsifier and 0.05-20% by weight of at least one flavor component,

wherein at least 30% of said composition is capable of reversing in phase within one minute after being introduced into water at 36°C.

12. (New) The composition as claimed in Claim 11, wherein a weight ratio of the aqueous phase to the oil phase ranges between 80:20 and 15:85.

13. (New) A water-in-oil emulsion composition comprising at least one member selected from the group consisting of fats, oils and mixtures thereof, said composition further comprising an aqueous phase based on water and an oil phase, said oil phase comprising at least 15% by weight of at least one diglyceride wherein said weight percent is based on a total weight of said oils, fats or mixtures thereof,

wherein at least 30% by weight of said composition is capable of reversing in phase within one minute after being introduced into water at 36°C.

REMARKS

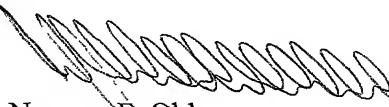
Claims 7-13 are active in this present application. Claims 1-6 have been cancelled.

Claims 7-13 are new claims. Support for the new claims is found in the original claims and on page 5, lines 1-20 and page 8, lines 17-23. No new matter is believed to have been added.

An action on the merits and allowance of claims is solicited.

Respectfully submitted,

OBLON, SPIVAK, McCLELLAND,  
MAIER & NEUSTADT, P.C.



Norman F. Oblon  
Attorney of Record  
Registration No. 24,618

Stefan U. Koschmieder, Ph.D  
Registration No. P 50,238



**22850**

(703) 413-3000

Fax #: (703)413-2220

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**Marked-Up Copy**

Serial No:

Amendment Filed on:

12-31-01

IN THE CLAIMS

Claims 1-6 (Cancelled).

Claims 7-13 (New).

Docket No. 216527US0PCT  
IN RE APPLICATION OF: Kenji MASUI, et al.  
SERIAL NO: NEW U.S. PCT APPLICATION BASED ON PCT/JP00/04359  
FILED: HEREWITH  
FOR: WATER-IN-OIL TYPE EMULSIFIED FAT AND/OR OIL COMPOSITION

ASSISTANT COMMISSIONER FOR PATENTS  
WASHINGTON, D.C. 20231

SIR:

Transmitted herewith is an amendment in the above-identified application.

- ☒ No additional fee is required
- ☐ Small entity status of this application under 37 C.F.R. §1.9 and §1.27 is claimed.
- ☒ Additional documents filed herewith: Declaration/Notice of Priority/Request for Consideration/PCT Transmittal Letter  
PCT/IB/304/Preliminary Amendment/International Search Report/PCT/IB/308  
International Preliminary Examination Report/Check for \$890.00

The Fee has been calculated as shown below:

CLAIMS	CLAIMS REMAINING		HIGHEST NUMBER PREVIOUSLY PAID	NO. EXTRA CLAIMS	RATE	CALCULATIONS	
TOTAL	7	MINUS	20	0	× \$18 =	\$0.00	
INDEPENDENT	3	MINUS	3	0	× \$84 =	\$0.00	
		<input type="checkbox"/> MULTIPLE DEPENDENT CLAIMS			+ \$280 =	\$0.00	
		TOTAL OF ABOVE CALCULATIONS					\$0.00
		<input type="checkbox"/> Reduction by 50% for filing by Small Entity					\$0.00
		<input type="checkbox"/> Recordation of Assignment			+ \$40 =	\$0.00	
		TOTAL					\$0.00

- ☐ A check in the amount of \$0.00 is attached.
- ☒ Please charge any additional Fees for the papers being filed herewith and for which no check is enclosed herewith, or credit any overpayment to deposit Account No. 15-0030. A duplicate copy of this sheet is enclosed.
- ☒ If these papers are not considered timely filed by the Patent and Trademark Office, then a petition is hereby made under 37 C.F.R. §1.136, and any additional fees required under 37 C.F.R. §1.136 for any necessary extension of time may be charged to Deposit Account No. 15-0030. A duplicate copy of this sheet is enclosed.

OBLON, SPIVAK, McCLELLAND,  
MAIER & NEUSTADT, P.C.

*Surinder Sachar*

Norman F. Oblon  
Registration No. 24,618

Surinder Sachar  
Registration No. 34,423



22850

Customer Number 22850  
Tel. (703) 413-3000  
Fax. (703) 413-2220  
(OSMMN 10/01)



10/019427

JC13 Rec'd PCT/PTO 31 DEC 2001

Docket No. 216527US0PCT

IN RE APPLICATION OF: Kenji MASUI, et al.

SERIAL NO: NEW U.S. PCT APPLICATION BASED ON PCT/JP00/04359

FILED: HEREWITH

FOR: WATER-IN-OIL TYPE EMULSIFIED FAT AND/OR OIL COMPOSITION

ASSISTANT COMMISSIONER FOR PATENTS  
WASHINGTON, D.C. 20231

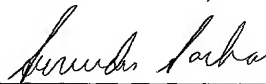
SIR:

Transmitted herewith is an amendment in the above-identified application.

- ☒ No additional fee is required
- ☐ Small entity status of this application under 37 C.F.R. §1.9 and §1.27 is claimed.
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International Preliminary Examination Report/Check for \$890.00

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TOTAL	7	MINUS	20	0	× \$18 =	\$0.00	
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		<input type="checkbox"/> MULTIPLE DEPENDENT CLAIMS			+ \$280 =	\$0.00	
		TOTAL OF ABOVE CALCULATIONS					\$0.00
		<input type="checkbox"/> Reduction by 50% for filing by Small Entity					\$0.00
		<input type="checkbox"/> Recordation of Assignment			+ \$40 =	\$0.00	
		TOTAL					\$0.00

☐ A check in the amount of **\$0.00** is attached.☒ Please charge any additional Fees for the papers being filed herewith and for which no check is enclosed herewith, or credit any overpayment to deposit Account No. 15-0030. A duplicate copy of this sheet is enclosed.☒ If these papers are not considered timely filed by the Patent and Trademark Office, then a petition is hereby made under 37 C.F.R. §1.136, and any additional fees required under 37 C.F.R. §1.136 for any necessary extension of time may be charged to Deposit Account No. 15-0030. A duplicate copy of this sheet is enclosed.OBLON, SPIVAK, McCLELLAND,  
MAIER & NEUSTADT, P.C.

Norman F. Oblon

Registration No. 24,618



22850

Customer Number 22850  
Tel. (703) 413-3000  
Fax. (703) 413-2220  
(OSMMN 10/01)

Surinder Sachar

Registration No. 34,423

## DESCRIPTION

## WATER-IN-OIL TYPE EMULSIFIED FAT AND/OR OIL COMPOSITION

## Technical Field

The present invention relates to a water-in-oil type emulsified fat and/or oil composition which comprises a diglyceride and which is excellent in a flavor release during the time for ingestion thereof. The water-in-oil type emulsified fat and/or oil composition of the present invention is preferably and suitably utilized in a margarine, a fat spread, a butter cream, a shortening, etc.

## Background Art

In recent years, the intake of lipid in eating habits in various developed countries is increasing to raise the concentration of triglycerides (neutral fat) in blood and to cause obesity, and this has been a major cause of Life style related disease.

Diglyceride has been revealed to be a fat or oil, after which is eaten, which hardly increases the concentration of a neutral fat in blood and hardly constitutes a body fat (JP-A 10-176181). Therefore, the diglyceride is expected to be used as the substitution of a conventional fat or oil in a (water-in-oil emulsion) food having a large amount of the fat or oil such as a

cooking oil, in particular, a margarine and a spread.

Various water-in-oil type emulsified compositions containing a diglyceride blended with a fat and/or oil are proposed, for example, in JP-B 7-121196, JP-A 3-91441, US-A 4284655, JP-A 61-63242, US-A 5879735, WO 95/22257, WO 96/32022 and the like, but there is no description of a flavor release from the flavor component thereof.

The present inventors have further continued in the investigation of the water-in-oil type emulsified fat and/or oil composition. As the result, they found the problem that the foods containing diglycerides in the high concentration during the time for ingestion and the foods are not good in flavor release related directly to the degree of good taste thereof. This phenomenon is not observed in a fat and/or oil being mainly based on a triglyceride.

#### Disclosure of the Invention

The present inventors have found that in a system of the water-in-oil type emulsion containing diglycerides in the high concentration, emulsification is so stable that the rate of the phase reversal of the emulsion from W/O type to O/W type in the mouth delays, thus affecting much of the flavor release.

The present invention provides a water-in-oil type emulsified fat and/or oil composition which is composed of (1) the aqueous phase based on

water and (2) the oil phase of fat and/or oil comprising 15 % by weight or more, based on the total oils and fats, of diglycerides, and the composition including a flavor component(s), 30 % by weight or more of the said emulsified composition being able to be reversed in phase within one minute after it has been introduced into water at 36 C°. Thus, the above-mentioned problem of the prior art has been solved.

Further, the fat(s) and/or oil(s) of the present invention comprise preferably 15 to 90 % by weight of diglycerides and 85 to 10 % by weight of triglycerides. The composition of the present invention may comprise 0.05 to 20 % by weight, preferably 0.1 to 10 % by weight, of flavor components.

#### Modes for Carrying Out the Invention

The fatty acid constituting the diglyceride used in the present invention includes a C<sub>6</sub> to C<sub>22</sub> saturated or unsaturated fatty acid, preferably a C<sub>16</sub> to C<sub>22</sub> unsaturated fatty acid. In the fatty acid group which is contained in the diglyceride, an unsaturated fatty acid is desirable in the amount of 55 % by weight or more.

As the starting fat or oil used in the diglyceride, a fat or oil containing a large amount of an unsaturated fatty acid-residue, for example, including a vegetable (or plant) fat or oil such as safflower oil, olive oil, cottonseed oil, rapeseed oil, corn oil, soybean oil and palm oil; further, an animal fat or oil such as lard, beef-tallow and butter; and a fractional oil thereof, a transesterified oil thereof and a hydrogenated oil (hardened oil) thereof may

be used. The diglyceride is obtained by ester-exchanging reaction (,ester-interchanging reaction or transesterification) of a mixture of one or more of these fats and oils and glycerol in the presence of a catalyst or by ester-exchanging reaction of a mixture of a fatty acid composition having the high levels of unsaturated fatty acids and glycerol in the presence of a catalyst.

An excess of monoglycerides formed in a mixture of the generated diglycerides can be removed by a molecular distillation method or a chromatography method. The remaining monoglycerides are desirably removed to make it as small as possible. When the content thereof is 5 % by weight or less (preferably 2 % by weight or less), there is no problem.

From the viewpoint of the stability in the emulsification, the content of diglycerides in the present invention is preferably 15 % by weight or more, particularly preferably 15 to 90 % by weight based on the amount of the fats and oils. The content is particularly preferably 45 % by weight or more from the viewpoint of controlling triglycerides in blood and inhibiting the accumulation of body fat.

The flavor component referred to in the present invention means all additives, related to tastes and aromas, such as perfumes, tasting agents (proteinous materials, amino acids etc.), spices, salts and sugars, and these are blended in an aqueous phase and/or an oil phase.

The degree of the phase reversal referred to in the present invention can be confirmed by means of the concentration of the flavor component blended in the aqueous phase in water, when 5 g of the emulsified fat and/or oil composition was dispersed in 50 g of water with a magnetic stirrer (with a bar of 2.5 cm in length, revolved at 300 rpm) at a controlled temperature of 36 °C in a beaker having its capacity of 100 ml. For example, in case a salt is placed as a flavor component, the degree of the phase reversal is represented in terms of the concentration of the salt in water after the emulsified composition have been dispersed in water at an oral temperature (about 36 °C) for one minute, given that the recognized salt concentration in water is made to 100 when demulsification is carried completely out by heating. If a sugar is placed as a flavor component, the concentration of the sugar may be measured by the same way as those in mentioned above. It is desired that 30 % or more, preferably 50 % or more and particularly preferably 80 % or more of the emulsion is made to reverse in phase for an initial one minute. When the degree of the phase reversal is less than 30 %, the strength of the flavor release is considerably low and the start of the release delays.

The composition exhibiting such a physical property can also be prepared even by selecting an ordinarily used formulation ingredient and an amount thereof. However, the demulsifier shown below (which is not ordinarily used in a W/O type emulsion) is

advantageously used in order to easily obtain such a composition.

The demulsifier referred to in the present invention is an additive functioning as a flavor enhancer or a flavor release-enhancer, which makes the phase reverse rapidly in the mouth during the time for ingestion, without affecting the stability in the emulsification during storage, to realize the excellent flavor release.

As the demulsifier, a polyglycerol fatty acid ester, a water-soluble decomposed protein, lysolecithin, a sucrose fatty acid ester, a monoglyceride organic acid ester and a sorbitan fatty acid ester described below may be used.

The polyglycerol fatty acid ester should be a mono-, di- or poly- ester of a saturated or unsaturated fatty acid having 10 to 22 carbon atoms, wherein the fatty acid constitutes the polyglycerol fatty acid ester, and polyglycerol and it should have HLB of 7 or more (preferably 11 or more). If the HLB is less than 7, the flavor release is worsened on the contrary.

As the decomposed protein, one or more kinds obtained by decomposing a milk protein, a vegetable (or plant) protein, an egg protein or the like with an enzyme or acid may be used. Specifically, the milk protein for use includes casein, lactalbumin, lactoglobulin, lactoferrin, whey (or milk serum), skim milk powder (or non-fat milk powder), whole milk powder, butter milk powder, milk serum protein, milk etc. The plant protein includes soybean

protein, wheat protein, corn protein etc. The egg protein includes ovalbumin, conalbumin, ovomucoid, ovoglobulin, egg white protein, yolk protein, whole egg protein etc.

As a preferable physical property of the decomposed protein obtained by decomposing one of these, water solubility is mentioned. If they are water-insoluble, the flavor release is worsened on the contrary due to gelation of the protein. Further, the decomposing treatment is carried preferably out before blending but it may be carried out during or after emulsification.

The lysolecithin is a water-soluble compound constituted from a fatty acid being a  $C_{10}$  to  $C_{22}$  saturated or unsaturated fatty acid and it has HLB of 8 or more (preferably 12 or more). If the HLB is less than 8, the flavor release is worsened on the contrary.

The sucrose fatty acid ester should be a mono-, di-, or poly-ester of a saturated or unsaturated fatty acid having 10 to 22 carbon atoms, wherein the fatty acid constitutes the sucrose fatty acid ester, and sucrose; and it should have HLB of 5 or more (preferably 8 or more). If the HLB is less than 5, the flavor release is worsened on the contrary.

The monoglyceride organic acid ester is one having 1 or 2 organic acids bonded to a monoglyceride constituted of a saturated or unsaturated fatty acid having 10 to 22 carbon atoms and it should have HLB of 8 or more. If the HLB is less than 8, the flavor release is worsened on the contrary.



The sorbitan fatty acid ester should be constituted from a saturated or unsaturated fatty acid having 10 to 22 carbon atoms, and it should have HLB of 8 or more. In particular, one of polysorbates known as Tween series from Atlas Ltd. in United States of America, including polyoxyethylene sorbitan monostearate (Tween 60), polyoxyethylene sorbitan tristearate (Tween 65), polyoxyethylene sorbitan monooleate (Tween 80), polyoxyethylene sorbitan monolaurate (Tween 20) and polyoxyethylene sorbitan monopalmitate (Tween 40) can be preferably used. Incidentally, if the HLB is less than 8, the flavor release is worsened on the contrary.

The value determined by the formula of Griffin was used as HLB of the polyglycerol fatty acid, monoglyceride organic acid ester or sorbitan fatty acid ester, while the value measured by the emulsification method was used as HLB of lysolecithin or the sucrose fatty acid ester.

It is important that the demulsifier is added in such an amount as to exhibit the action of demulsification (effect of improving the flavor release). Although the demulsifier is preferably added to the aqueous phase, there is no problem if it is added to the oil phase. Its approximate amount is within the range of 0.01 to 5 % by weight, preferably 0.1 to 1 % by weight, in the composition.

The starting material of an edible fat or oil used in the

present invention may be one or more selected from vegetable fats and oils including soybean oil, rapeseed oil, palm oil, corn oil, cottonseed oil, coconut oil and palm kernel oil; and animal fats and oils including lard, fish oil and milk fat; and those obtained by hydrogenation or ester-exchange of these oils, for use.

The solid fat content (SFC) in the oil phase used in the present invention may be usually within the range of 1 to 35 at 5 to 35 °C, preferably 7 to 30 at 20 °C and preferably 1 to 20 at 30 °C.

The water-in-oil type emulsified fat and/or oil composition of the present invention may be prepared in a usual manner, and the ratio of the aqueous phase based on water to the oil phase by weight can be made within the range of the oil phase : the aqueous phase = 20 : 80 to 85 : 15 (preferably 40 : 60 to 85 : 15).

A dairy product, an emulsifier or the like as a sub-component in the oil phase may be blended with the composition of the present invention. Further, milk protein, starch, a thickening polysaccharide, a thermoplastic protein (gelatin etc.) or the like may be blended with the aqueous phase based on water.

Further, an anti-oxidant including tocopherol, an ascorbyl ester such as palmitate and stearate, a tea leaf, a herb such as rosemary, a natural anti-oxidizing component extracted from a leaf or a root of a peach may be used together in order to inhibit the deterioration of the water-in-oil type emulsified fat and/or oil

composition.

## Examples

### [Preparation of the diglyceride]

While the fatty acid obtained from a soybean oil (obtained by wintering at 0 °C) and glycerol were mixed at the molar ratio of 2 : 1, they were reacted at 70 °C for 3 hours in the presence of a catalyst as a commercial lipase preparation of immobilized 1,3-position-selective lipase (Lipozyme 3A™ provided by Novo Industry A. S.). During the reaction, the inside pressure of the system was reduced to 0.26 kPa in order to remove water formed by esterification. After the lipase preparation was removed by filtration from the reaction product, the filtrate was subjected to molecular distillation, decoloration and deodorization to obtain the diglyceride (consisting of diglyceride content of 85 % by weight, monoglyceride content of 1.3 % by weight and triglyceride content of 13.7 % by weight and having open tube melting point of 5 °C).

### Examples 1 to 5 and Comparative Examples 1 to 4

According to the recipe of Table 1, the compositions were emulsified at 70 °C for 10 minutes by a homogenizing mixer (Tokushu Kika Kogyo Co., Ltd.) to make the plastic emulsion 800 g after the reaction. The obtained emulsion was rapidly cooled and plasticized in a usual manner whereby a water-in-oil type

emulsified fat and/or oil composition was produced.

The degree of phase reversal of the obtained water-in-oil type emulsified fat and/or oil composition was measured in the following manner.

Namely, after 50 g of water was placed in a beaker having its capacity of 100 ml and was adjusted to 36 °C, 5 g of the emulsified fat and/or oil composition was dispersed therein with a magnetic stirrer (with a bar of 2.5 cm in length, revolved at 300 rpm). The salt concentration in the water was measured with time. The degree of phase reversal was made as  $(\text{salt concentration in water after one minute}) \div (\text{salt concentration upon complete demulsification}) \times 100$ . Then, a compact meter C-121 (Horiba, Ltd.) for a salt content was used for the measurement of the salt concentration. The results are shown in Table 1.

Then, the obtained water-in-oil type emulsified fat and/or oil compositions (the product just after preparation and the product after storage at 5 °C for 30 days) were evaluated in viewpoint of their flavor release upon eating in mouth under the following criteria by special panelists (10 panelists). The average points are also shown in Table 1.

The evaluation criteria are as follows.

- 4: The flavor release is very good.
- 3: The degree of the flavor release is slightly weak and the start of the release delays slightly.

2: The degree of the flavor release is extremely weak and the start of the release delays.

1: The degree of the flavor release is extremely bad and the start of the release delays extremely.

Table 1

	Examples					Comparative Examples			
	1	2	3	4	5	1	2	3	4
Oil phase (parts)	Diglyceride	48.9	48.9	48.9	55.5	48.9	48.6	48.6	48.9
	Partially hydrogenated (hardened) palm oil (IV = 40)	21.0	21.0	21.0	13.9	21.0	20.8	20.8	21.0
	Stearic acid monoglyceride	—	—	—	—	—	—	—	—
	Additive 1 type	—	—	—	—	—	F	G	—
	Vitamin E	0.02	0.02	0.02	0.02	0.02	0.02	0.5	0.02
	$\beta$ -Carotene	0.002	0.002	0.002	0.002	0.002	0.002	0.02	0.002
Aqueous phase (parts)	Butter flavor	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
	Cheese flavor	—	—	—	—	—	—	—	—
	Distilled water	27.9	27.9	27.9	27.9	27.9	28.4	28.4	28.4
	Additive 2 type	A	B	C	D	E	—	—	—
	Skim milk powder	0.5	0.5	0.5	0.5	0.5	—	—	—
	Common salt	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3
Degree of phase reversal (%)	Fresh cream	1.3	1.3	1.3	1.3	1.3	1.3	1.3	1.3
		—	—	—	—	—	—	—	—
Flavor release (just after production)		100	100	100	100	17	17	8	25
		4	4	3.9	3.5	1.2	1	1	2
		4	4	3.9	3.5	1	1	1	2

SFC in the oil phase in Examples 1 to 4 and Comparative Examples 1 to 4 was 15.3 at 20 °C or 8.8 at 30 °C; and SFC in the oil phase in Example 5 was 9.8 at 20 °C or 6.0 at 30 °C.

- A: Decomposed whey protein (provided by Morinaga Milk Industry Co., Ltd.)
- B: Sucrose fatty acid ester having HLB of 15 (F-160, provided by Dai-ichi Kogyo Seiyaku Co., Ltd.)
- C: Polyglycerol fatty acid ester having HLB of 15 (ML-750, provided by Sakamoto Yakuhin Kogyo Co.; Ltd.)
- D: Polyglycerol fatty acid ester having HLB of 11 (SO-750, provided by Sakamoto Yakuhin Co., Ltd.)
- E: Whey protein (provided by Morinaga Milk Industry Co., Ltd.)
- F: Sucrose fatty acid ester having HLB of 1 (F-10, provided by Dai-ichi Kogyo Seiyaku Co., Ltd.)
- G: Polyglycerol fatty acid ester having HLB of 4.5 (Sunsoft Q-175S, provided by Taiyo Kagaku Co., Ltd.)

## CLAIMS

1. (Amended) Use of a demulsifier to reverse at least 30% of the w/o phases of a composition comprising (1) the aqueous phase based on water and (2) the oil phase of fat and/or oil comprising 15 % by weight or more, based on the total oils and fats, of diglycerides and a flavor component(s) within one minute after it has been introduced into water at 36 °C.
2. (Amended) The use as claimed in Claim 1, in which the fat and/or oil comprises 15 to 90 % by weight of diglycerides and 85 to 10 % by weight of triglycerides and 0.05 to 20 % by weight of the flavor component and 0.01 to 5 % by weight of the demulsifier are included in the composition.
3. (Amended) The use as claimed in Claim 1 or 2, in which the weight ratio of the aqueous phase to the oil phase ranges between 80:20 and 15:85.
4. (Amended) The use as claimed in Claim 1 or 2, in which the demulsifier is at least one member selected from the group consisting of a polyglycerol fatty acid ester having HLB of 7 or more, a water-soluble decomposed protein, lysolecithin having HLB of 8 or more, a sucrose fatty acid ester having HLB of 5 or more, a monoglyceride organic acid ester having HLB of 8 or more, and a sorbitan fatty acid ester having HLB of 8 or more.
5. (Amended) A water-in-oil emulsion composition, comprising



(1) the aqueous phase based on water, (2) the oil phase of fat and/or oil comprising 15 to 90 % by weight, based on the total oils and fats, of diglycerides and 85 to 10 % by weight of triglycerides, 0.01 to 5 % by weight of a demulsifier and 0.05 to 20 % by weight of a flavor component(s), at least 30% of the composition being able to be reversed in the phases within one minute after it has been introduced into water at 36 °C.

6. (Amended) The composition as claimed in Claim 5, in which the weight ratio of the aqueous phase to the oil phase ranges between 80:20 and 15:85.

## Declaration and Power of Attorney For Patent Application

### 特許出願宣言書及び委任状

### Japanese Language Declaration

### 日本語宣言書

下記の氏名の発明者として、私は以下の通り宣言します。

As a below named inventor, I hereby declare that:

私の住所、郵便の宛先、国籍は下記の私の氏名の後に記載された通りです。

My residence, mailing address and citizenship are as stated next to my name.

下記の名称の発明に関して請求範囲に記載され、特許出願している発明内容について、私が最初かつ唯一の発明者（下記の氏名が一つの場合）もしくは最初かつ共同発明者（下記の名称が複数の場合）であると信じています。

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled.

上記発明の明細書は、

WATER-IN-OIL TYPE EMULSIFIED FAT AND/OR OIL COMPOSITION

the specification of which

☐ 本書に添付されています。

☒ is attached hereto.

☐ 月 日に提出され、米国出願番号または特

☒ was filed on June 30, 2000

許協定条約国際出願番号を

as United States Application Number or PCT International Application Number

とし、

PCT/JP00/04359 and was amended on

(該当する場合) に訂正されました。

(if applicable)

私は、特許請求範囲を含む上記訂正後の明細書を検討し、内容を理解していることをここに表明します。

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

私は、連邦規則法典第37編第1条56項に定義されるとおり、特許資格の有無について重要な情報を開示する義務があることを認めます。

I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, § 1.56.

# Japanese Language Declaration (日本語宣言書)

私は、米国法典第35編119条(a) - (d)項又は365条 (b) 項に基づき下記の、米国以外の国の少なくとも一カ国を指定している特許協力条約365(a)項に基づく国際出願、又は外国での特許出願もしくは発明者証の出願についての外国優先権をここに主張するとともに、優先権を主張している、本出願の前に出願された特許または発明者証の外国出願を以下に、枠内をマークすることで、示しています。

Prior Foreign Application(s)  
外国での先行出願

11-184762

(Number)  
(番号)

Japan

(Country)  
(国名)

I hereby claim foreign priority under Title 35, United States Code, § 119 (a)-(d) or 365(b) of any foreign application(s) for patent or inventor's certificate, or § 365(a) of any PCT International application which designated at least one country other than the United States, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or PCT International application having a filing date before that of the application on which priority is claimed.

Priority Claimed  
優先権主張

30 June 1999

(Day/Month/Year Filed)  
(出願年月日)

☒

Yes  
はい

☐

No  
いいえ

私は、第35編米国法典119条 (e) 項に基づいて下記の米国特許出願規定に記載された権利をここに主張いたします。

(Application No.)  
(出願番号)

(Filing Date)  
(出願日)

I hereby claim the benefit under Title 35, United States Code, §119(e) of any United States provisional application(s) listed below.

(Application No.)  
(出願番号)

(Filing Date)  
(出願日)

私は、下記の米国法典第35編120条に基づいて下記の米国特許出願に記載された権利、又は米国を指定している特許協力条約365条 (c) に基づく権利をここに主張します。また、本出願の各請求範囲の内容が米国法典第35編112条第1項又は特許協力条約で規定された方法で先行する米国特許出願に開示されていない限り、その先行米国出願書提出日以降で本出願書の日本国内または特許協力条約国際提出日までの期間中に入手された、連邦規則法典第37編1条56項で定義された特許資格の有無に関する重要な情報について開示義務があることを認識しています。

I hereby claim the benefit under Title 35, United States Code, § 120 of any United States application(s), or § 365(c) of any PCT International application designating the United States, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of Title 35, United States Code, § 112, I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, § 1.56 which became available between the filing date of the prior application and the national or PCT International filing date of this application.

PCT/JP00/04359

(Application No.)  
(出願番号)

June 30, 2000

(Filing Date)  
(出願日)

(Status: Patented, Pending, Abandoned)  
(現況：特許許可済、係属中、放棄済)

(Application No.)  
(出願番号)

(Filing Date)  
(出願日)

(Status: Patented, Pending, Abandoned)  
(現況：特許許可済、係属中、放棄済)

私は、私自身の知識に基づいて本宣言書中で私が行なう表明が真実であり、かつ私の入手した情報と私の信じていること、さらに故意になされた虚偽の表明及びそれと同等の行為は米国法典第18編第1001条に基づき、罰金または拘禁、もしくはその両方により処罰されること、そしてそのような故意による虚偽の声明を行なえば、出願した、又は既に許可された特許の有効性が失われることを認識し、よってここに上記のごとく宣誓を致します。

委任状：私は下記の発明者として、本出願に関する一切の手続きを米特許商標局に対して遂行する弁理士または代理人として、下記の者を指名いたします。  
(弁理士、または代理人の指名及び登録番号を明記のこと)

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith: (list name and registration number)

Japanese Language Declaration  
(日本語宣言書)



022850

書類送付先

Send Correspondence to:



022850

直接電話連絡先：(名前及び電話番号)

Direct Telephone calls to: (name and telephone number)

(703) 413-3000

単独発明者または第一の共同発明者の氏名	1-00	Full name of sole or first inventor	Kenji Masui
発明者の署名	日付	Inventor's signature	Date Dec 6, 2001
住所		Residence	c/o Kao Corporation, Research Laboratories, 2-1-3, Bunka, Sumida-ku, Tokyo 131-8501, Japan JPX
国籍		Citizenship	Japan
郵便の宛先		Mailing Address	same as above

第二の共同発明者の氏名	2-00	Full name of second joint inventor, If any	Yoshihiro Konishi
第二の共同発明者の署名	日付	Second inventor's signature	Date Dec 10, 2001
住所		Residence	c/o Kao Corporation, Research Laboratories, 1334, Minato, Wakayama-shi, Wakayama 640-8580, Japan JPX
国籍		Citizenship	Japan
郵便の宛先		Mailing Address	same as above